DEVELOPMENT OF A NOVEL LASER MATERIAL FOR MINIATURIZED LASER SYSTEMS

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Principal Investigator: Dr. Walter Zwicker (914) 762-0300

Monitored by: James Gibson

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The goals and duration of this program have been changed so as to complete the technical effort by the end of the year. This acceleration of effort is to provide experimental data on laser materials of neodymium pentaphosphate (NPP) and also stoichiometric erbium, the latter being a new requirement. This data is required by NV&EOL to initiate near-term development of a miniature 1.06 μm laser marker and an eyesafe, miniature rangefinder. Our work during this quarter dealt with the growth and evaluation of NPP crystals for a 1.06 μm miniature laser and Er^{3+} stoichiometric crystals for an eyesafe

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20. ABSTRACT (Cont'd.)

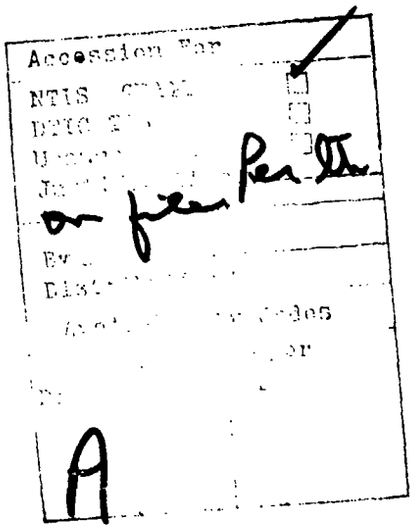
miniature laser emitting at 1.54 μm. Crystals of NPP up to 2x2x20 mm for fabrication of miniature laser rods were grown by a previously described high-temperature, solution-growth technique. Flash-lamp-pumped lasing experiments were conducted using various sizes of NPP crystals, in both the free-oscillation and Q-switched modes at different pulse repetition rates. Output pulse energies and properties of the laser output beam were measured. The efficiencies for the free-oscillation mode were close to the maximum published values; for the Q-switched mode, the efficiencies were low compared to published values. Crystals of ErP_5O_{14} were grown by the same technique used for NPP; crystals of $ErLiP_4O_{12}$ and $ErAl_3(BO_3)_4$ were grown by related fluxed-melt techniques. These crystals were several millimeters in size. Both Yb and Gd were added in various ratios as sensitizing and/or diluent ions. The absorption and emission properties of the Er^{3+} luminescence in some samples of ErP_5O_{14}, $LiErP_4O_{12}$, and $ErAl_3(BO_3)_4$ have been studied both in the visible and infrared regions of the spectrum. Preliminary evaluations of these materials for laser applications have been made.

PREFACE

This work is being performed by Philips Laboratories, a Division of North American Philips Corporation, Briarcliff Manor, New York under the overall supervision of Dr. Rameshwar Bhargava, Director, Exploratory Research Group. Dr. Walter Zwicker, Senior Program Leader for Crystal Growth and Materials Technology, is the Principal Investigator. Mr. Emil Abelaf and Mr. Theodore Kovats are responsible for crystal growing; Dr. Sel Colak and Mr. Jacob Khurgin are responsible for materials evaluation as well as laser design and construction.

This program is sponsored by the Defense Advanced Research Projects Agency (DARPA) and was initiated under Contract No. MDA903-81-C-0034. James Gibson is the Contracting Officer's Technical Representative for DARPA.

The work described in this fourth Quarterly Technical Report covers the period from 1 July to 30 September 1981.



SUMMARY

The goals and duration of this program have been changed so as to complete the technical effort by the end of the year. This acceleration of effort is to provide experimental data on laser materials of neodymium pentaphosphate (NPP) and also stoichiometric erbium, the latter being a new requirement. This data is required by NV&EOL to initiate near-term development of a miniature 1.06 μm laser marker and an eyesafe, miniature rangefinder. Our work during this quarter dealt with the growth and evaluation of NPP crystals for a 1.06 μm miniature laser and Er^{3+} stoichiometric crystals for an eyesafe miniature laser emitting at 1.54 μm. Crystals of NPP up to 2x2x20 mm for fabrication of miniature laser rods were grown by a previously described high-temperature, solution-growth technique. Flash-lamp-pumped lasing experiments were conducted using various sizes of NPP crystals, in both the free-oscillation and Q-switched modes at different pulse repetition rates. Output pulse energies and properties of the laser output beam were measured. The efficiencies for the free-oscillation mode were close to the maximum published values; for the Q-switched mode, the efficiencies were low compared to published values. Crystals of ErP_5O_{14} were grown by the same technique used for NPP; crystals of $ErLiP_4O_{12}$ and $ErAl_3(BO_3)_4$ were grown by related fluxed-melt techniques. These crystals were several millimeters in size. Both Yb and Gd were added in various ratios as sensitizing and/or diluent ions. The absorption and emission properties of the Er^{3+} luminescence in some samples of ErP_5O_{14}, $LiErP_4O_{12}$, and $ErAl_3(BO_3)_4$ have been studied both in the visible and infrared regions of the spectrum. Preliminary evaluations of these materials for laser applications have been made.

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1. INTRODUCTION

The goals and duration of this program have been changed substantially during this quarter. All technical effort is to be completed by the end of this year, and will be concentrated on evaluation of the lasing properties of neodymium pentaphosphate, as well as on the crystal growth and optical evaluation of stoichiometric erbium compounds for miniature lasers emitting in the eyesafe region of the spectrum. This latter effort is a new requirement.

Crystal growth and fabrication of miniature laser rods of neodymium pentaphosphate have continued on a routine basis, while crystals of erbium pentaphosphate, erbium-lithium tetraphosphate and erbium-aluminum borate were grown for the first time. Gadolinium or ytterbium were added to most of the erbium crystals as sensitizing ions.

Lasing experiments on neodymium pentaphosphate have been done with different flash-lamps and cavities, and the output behavior of these lasers was also studied with a passive Q-switch material. In these experiments, we observed laser output pulse energies up to 180 mJ in the free oscillation mode and up to 30 mJ in the Q-switched mode. We believe these results can be further improved by optimizing the output coupling of the lasers. In separate experiments, we operated the laser at a rate of 5 pps and observed that the output degraded, most probably due to thermally induced effects. The best beam divergence was about 2 mrad.

Initial characterization of ErP_5O_{14}, $LiErP_4O_{12}$, and $ErAl_3(BO_3)_4$ has been done. During these studies, the absorption and emission spectra of the samples were obtained. Some of the samples contained either Yb or Gd for energy transfer and/or dilution purposes.

2. MATERIAL PREPARATION AND CRYSTAL GROWTH

2.1 NdP_5O_{14} (NPP)

Growth of NPP crystals, up to 25 mm in cross section has continued on a routine basis. Some of the crystals had feathery-shaped inclusions 2-3 mm in size. The formation of these inclusions was correlated with the cracking of the quartz chamber holding the vitreous carbon crucible with the melt. This occurred numerous times, usually during the later part of the extended (3 weeks) growth run, and was caused by devitrification of, and attack on, the quartz by phosphric acid vapors. To overcome this problem, we have started experimental runs to remove these acid vapors by using a flow of a dry, inert gas, viz., nitrogen.

Fabrication of laser rods from NPP crystals is known to be difficult (Ref. 1). The main problem is "scaling" of the "b" face during final cutting of the laser rods from plates having optically polished "c" faces. The scaling is particularly noticeable toward the edges. Experiments with a new type of "multi-wafer" saw have been started to reduce or eliminate this problem, which effects the optical pumping efficiency of the rod.

2.2 $Er_xGd_{1-x}P_5O_{14}$ and $Er_xYb_{1-x}P_5O_{14}$

Crystals of compounds of both of these series were grown by basically the same technique described previously for the growth of NPP crystals (Ref. 1). However, since growth temperature solubility and even crystal structure for the resulting product will change with changing effective radius of the rare earth ions (see Fig. 1), optimum growth conditions have to be established for each individual compound.

ErP_5O_{14} crystals required a solution three times as concentrated as that for NPP crystals (i.e., a mass fraction of ~ 10% instead of 3.5% of R.E. oxide). The growth temperature, not yet optimized, was 680°C, which is about 100°C higher than the growth temperature for NPP crystals. The resulting crystals were mostly prismatic or hexagonal shaped and averaged about 1 x 2 x 3 mm (see Fig. 2).

$Er_{0.5}Yb_{0.5}P_5O_{14}$ crystals, grown under identical conditions, were of similar shape, with an average size of 2 x 2 x 5 mm but not free of some inclusions (see Fig. 3).

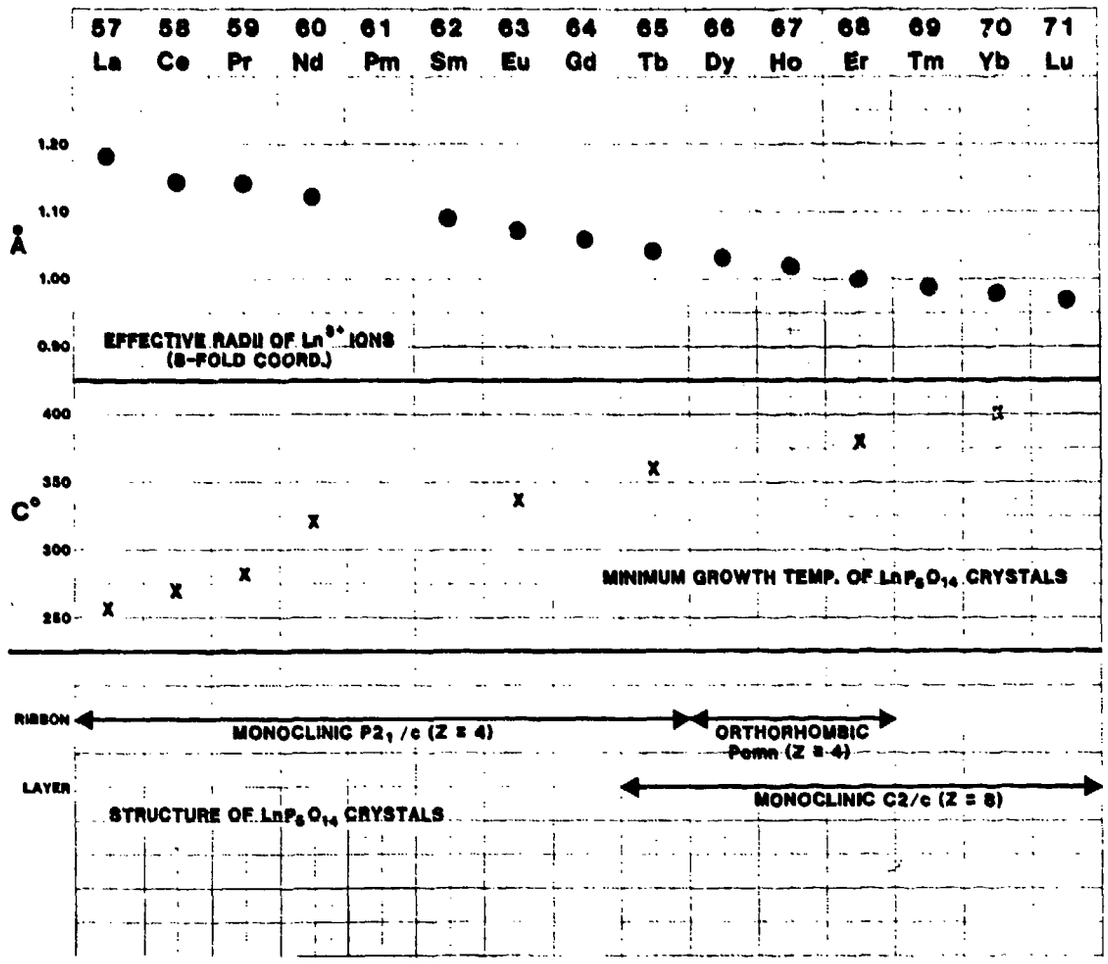


Figure 1: Effective ionic radii of rare-earth ions as well as minimum growth temperatures and crystal structures of corresponding pentaphosphate crystals of these rare earths.

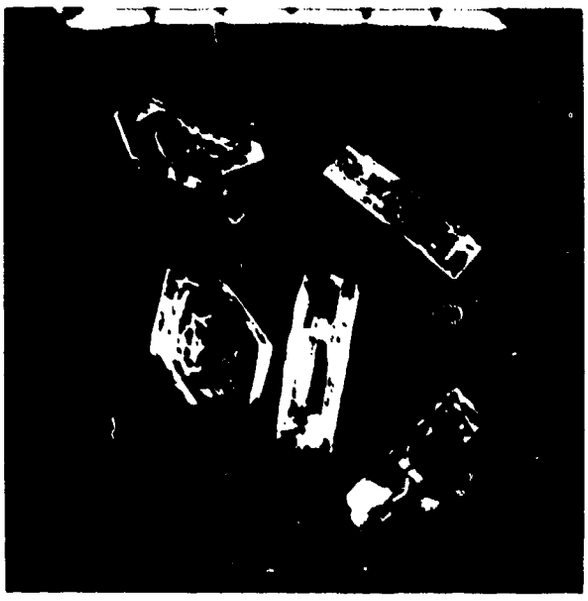


Figure 2: Crystals of ErP_5O_{14} (scale in mm).

Figure 3. Crystals of $Er_{0.5}Yb_{0.5}P_5O_{14}$ (magnification 10X).

$Er_xGd_{1-x}P_5O_{14}$ crystals with x of 0, 0.3, 0.5 and 0.7 were all grown, again, under similar conditions. The crystals were prismatic or hexagonal shaped, and up to 2 x 2 x 5 mm in size. Many contained inclusions in their centers (see Fig. 4).

Figure 4: Crystals of $Er_xGd_{1-x}P_5O_{14}$ (scale in mm).

2.3 $Er_xGd_{1-x}LiP_4O_{12}$ and $Er_xYb_{1-x}LiP_4O_{12}$

A series of crystal growth runs were made using the compositions listed in Table 1. The flux was held at 800°C for 16 hours and cooled 1°/hour to 700°C. The run was then air-quenched to 500°C and cooled in a furnace at 100°C/hour to room temperature. These runs were similar to the flux growth runs done on LNP.

Erbium or ytterbium and erbium-containing crystals tended to be needle-like while gadolinium and erbium-containing crystals tended to show a short prismatic morphology. All of these crystals suffer from a a bubble-like feature on the surface (see Fig. 5) which may be due to the flux-dissolving back material. In addition, most of the crystals show various types of inclusions.

Table 1

Flux Composition (Mol %)	Anticipated Composition of Crystal
3.5 Er_2O_3, 44 Li_2O, 52.5 P_2O_5	$ErLiP_4O_{12}$
1.75 Gd_2O_3, 1.75 Er_2O_3, 44 Li_2O, 52.5 P_2O_5	$Gd_{0.5}Er_{0.5}P_4O_{12}$
1.17 Gd_2O_3, 2.33 Er_2O_3, 44 Li_2O, 52.5 P_2O_5	$Gd_{0.34}Er_{0.66}P_4O_{12}$
1.75 Gd_2O_3, 1.75 Er_2O_3, 44 Li_2O, 52.5 P_2O_5	$Gd_{0.5}Er_{0.5}P_4O_{12}$
1.75 Yb_2O_3, 1.75 Er_2O_3, 44 Li_2O, 52.5 P_2O_5	$Yb_{0.5}Er_{0.5}P_4O_{12}$
3.5 Gd_2O_3, 44 Li_2O, 52.5 P_2O_5	$GdLiP_4O_{12}$
3.5 Y_2O_3, 44 Li_2O, 52.5 P_2O_5	$YLiP_4O_{12}$
0.35 Gd_2O_3, 0.35 Er_2O_3, 2.8 Y_2O_3, 44 Li_2O, 52.5 P_2O_5	$Y_{0.8}Gd_{0.1}Er_{0.1}P_4O_{12}$

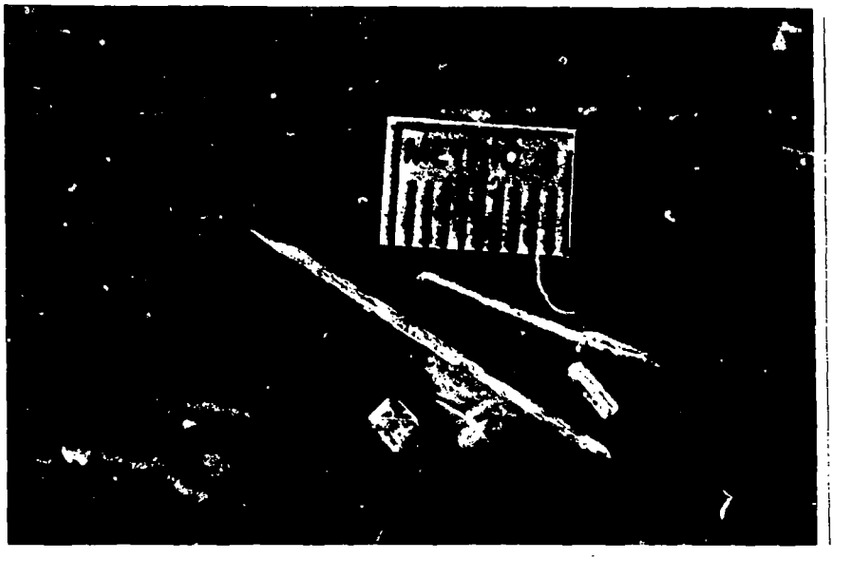


Figure 5: Crystals of $Er_xGd_{1-x}Li(PO_3)_4$ (short prismatic) and $Er_xYb_{1-x}Li(PO_3)_4$ (needles).

Growth experiments were also performed on $YLiP_4O_{12}$ (YLP), a possible new laser host. Very clear platelets of $YLi(PO_3)_4$ (Fig. 6, left) and $Y_{0.8}Gd_{0.1}Er_{0.1}P_4O_{12}$ (Fig. 6, right) were grown. These preliminary experiments show that, under the same growth condition, YLP with erbium and gadolinium produces crystals of better optical quality.

Table 1 also lists the anticipated, approximate composition of the crystals obtained from these runs. These crystals are presently being chemically analyzed to determine their exact composition.

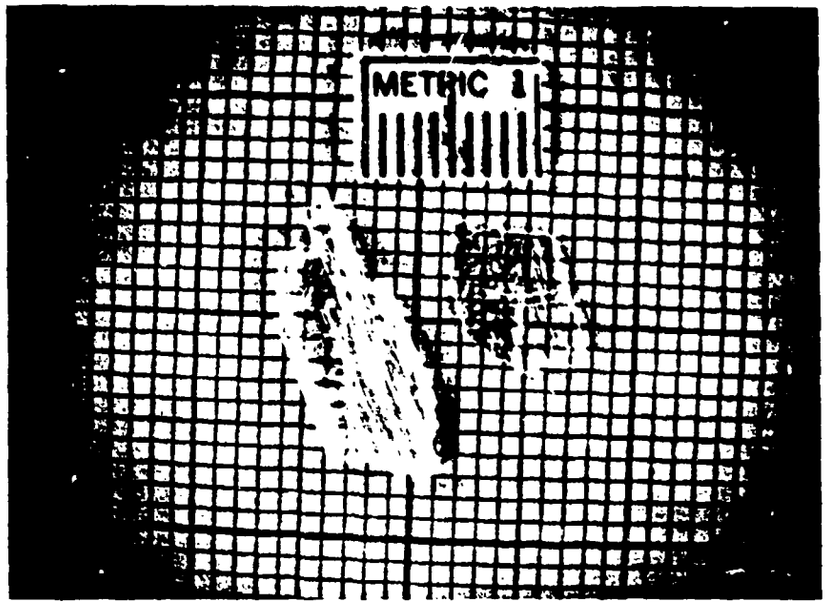


Figure 6: Crystals of $YLi(PO_3)_4$ (left) and $Y_{0.8}Gd_{0.1}Er_{0.1}(PO_3)_4$ (right).

2.4 $Er_xYb_{1-x}Al_3(BO_3)_4$ and $Er_xGd_{1-x}Al_3(BO_3)_4$

Crystals of these compounds were grown by a fluxed melt technique described previously (Ref. 2) for the growth of NAB crystals. The composition of the melt was:

76.32 g	PbO
95.52 g	B_2O_3
10.65 g	Gd_2O_3 or Yb_2O_3
21.82 g	Al_2O_3
16.85 g	Er_2O_3

Although the crystals were up to 15 mm in cross section, they contained very few clear areas. Preliminary experiments on the growth of such crystals from a Li_2O–B_2O_3 flux were unsuccessful since the flux did not clear even at elevated temperatures.

3. NdP_5O_{14} LASER EXPERIMENTS

Various sizes of NPP crystals were used in flash-lamp-pumped laser experiments to evaluate the performance of this material in free oscillation and Q-switched modes. Three types of flash lamps were used: Type 1 was the low efficiency type mentioned in our earlier reports; Type 2 was supplied to us by NV&EOL; Type 3 was obtained from Lincoln Laboratories. With these lamps and different cavity configurations, we performed the laser experiments and found that some of the laser ouput requirements were satisfied in some of the experiments. These results are given in the following paragraphs. Presently, we are improving the experimental conditions in order to obtain all of the desired properties in a single final setup.

The performances of the flash lamps obtained from NV&EOL and MIT Lincoln Laboratories (Types 2 and 3) were similar. A typical output vs. input characteristic obtained from a 2x2x20 mm crystal is shown in Figure 7; this is similar to that obtained previously by Chinn and Zwicker (Ref. 3). We were not able to reproduce the results reported later by Chinn (Ref. 4) who used a fourth type of flash lamp similar to Type 3 but with a better efficiency. Chinn was able to obtain about 35 mJ laser output energy for 2 J of electrical input energy with that flash lamp.

A second experiment was done by using, again, a 2x2x20 mm crystal but with a Type 3 flash lamp in a cavity with nearly flat mirrors, with a goal to improve the far-field beam properties. In this experiment, the output energies were lower (see Fig. 8) compared to those of the first experiment described above, due to increased sensitivity in alignment of nearly flat mirrors. With a better alignment procedure, the output energies can be made to exceed the output energies for the nearly confocal case described above. With the present cavity with nearly flat mirrors, we obtained a single transverse mode output with a 6 mm spot size at 3 meters distance from the laser. This corresponds to a far-field beam angle of about 2 mrad. We have also tried Q-switching with a standard Eastman Q-switch acetate sheet (Cat. No. 15064) in the same cavity configuration. The Q-switched output energies were about or less than 1 mJ for 2 J of input electrical energy. This corresponds to a Q-switch efficiency of about 10-20%, which is very low.

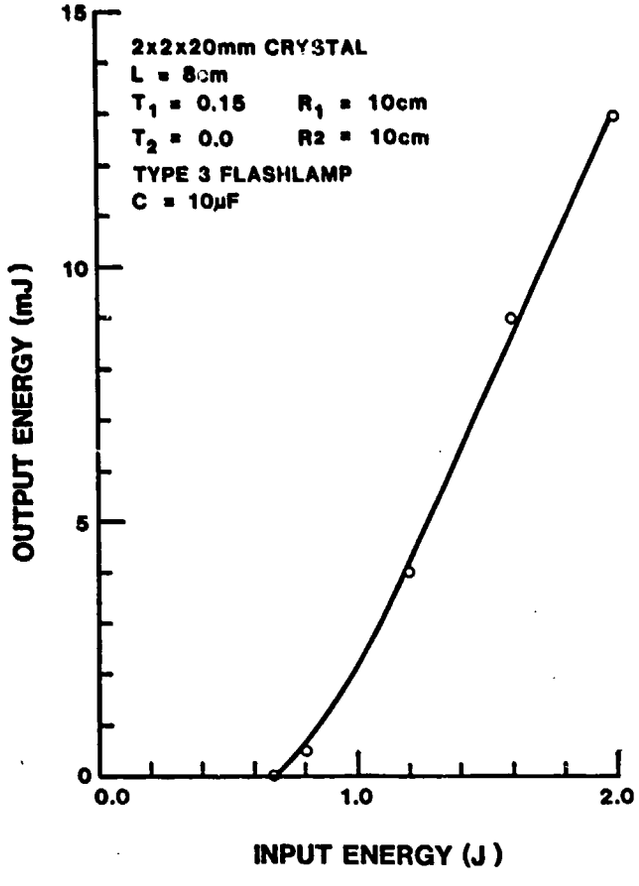


Figure 7: Typical output energy vs. input energy for a 2x2x20 mm NPP crystal. Although results are with a Type 3 flash lamp (see text), Type 2 flash lamp has similar behavior.

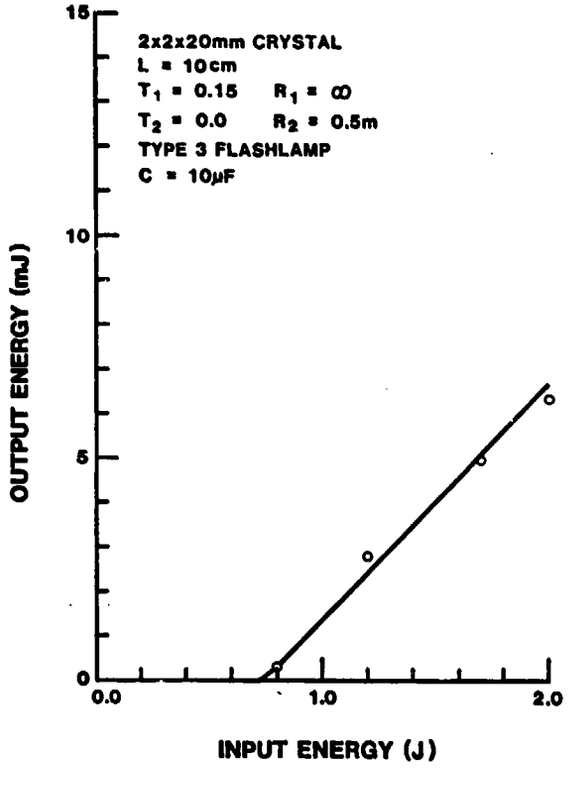


Figure 8: Output energy vs. input energy for a 2x2x20 mm NPP crystal in a nearly flat mirror cavity.

Another test was done with a 2x2x10 mm NPP crystal which was HR coated on one face. The output energies obtained with a Type 3 flash lamp (10 mm arc length) are given in Figure 9.

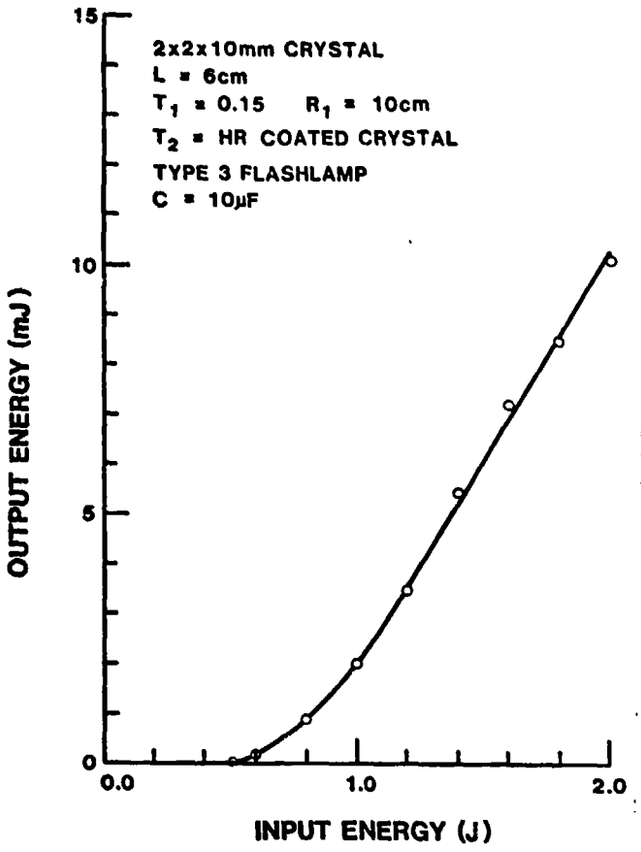
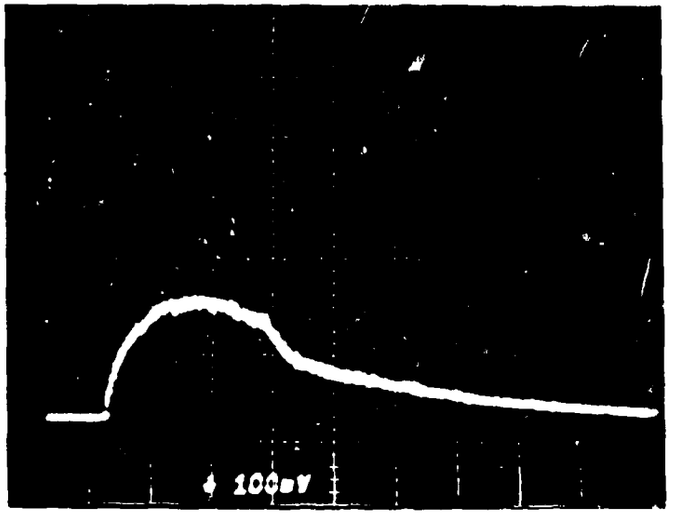


Figure 9: Output energy vs. input energy of a 2x2x10 mm NPP crystal HR coated on one face.

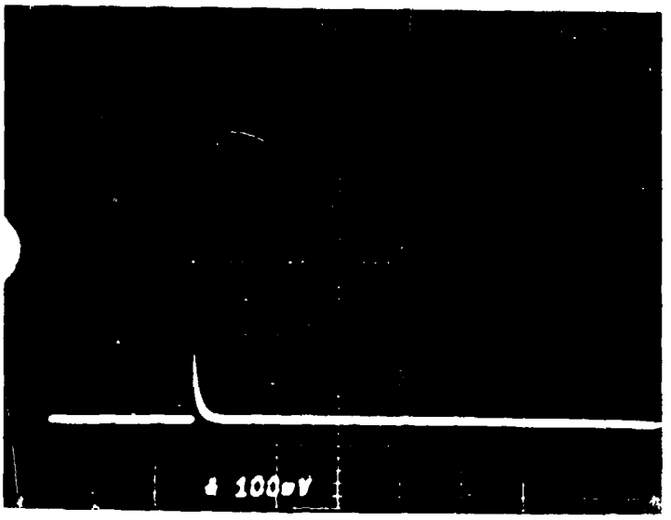
To achieve higher output energies in the Q-switched mode, we used Type 1 flash lamps to which we could apply more electrical input energy. Although these lamps are less efficient, they could go to high energies without failing, thereby improving the highest obtainable output energy from the laser. The output energies obtained with such a lamp in both the Q-switched and free oscillation modes are given in Table 2. The shapes of the laser output pulses for these modes are shown in Figure 10. Although the Q-switched output energies are high, the efficiencies are still between 10-20%.

Table 2: Q-Switching Output Energies

Input Energy (J)	Output Energy (mJ)	
	Q-Switched	Free Oscillation
75	30	180
59	17	110



(a) Q-switched mode (100 μs/div).



(b) Free-oscillation mode (100 μs/div).

Conditions

2x2x20 NPP crystal
L = 8 cm
T_1 = 0.04 R_1 = 10 cm
T_2 = HR R_2 = 10 cm
Type 1 flash lamps
C = 1300 F

Figure 10: Pulse shapes of laser output in Q-switched and free oscillation modes. Input electrical energy to flash lamp was 75 J. Maximum output energies were 180 mJ and 30 mJ for free-oscillation and Q-switched modes, respectively.

In another set of experiments, we tried different pulse repetition rates in a cavity using a Type 3 flash lamp energized by a 10 μF capacitor. It was possible to operate this NPP laser in the free oscillation mode at rates around 5 pps. However, the output energy of the laser degraded as a function of time, reaching zero output after about 20 pulses. The output of the laser recovered after a few seconds. We checked the potential across the capacitor during the pulses and found that it stayed approximately constant. This shows that the degradation in the output energy is most probably due to ground state absorption because of heating.

4. PRELIMINARY OPTICAL EVALUATION OF $Er_xM_{1-x}P_5O_{14}$, $LiEr_xP_4O_{12}$, AND $Er_xM_{1-x}Al_3(BO_3)_4$ CRYSTALS (M=Yb,Gd)

Preliminary optical evaluations were performed on crystals of $Er_xM_{1-x}P_5O_{14}$, $LiEr_xM_{1-x}P_4O_{12}$, and $Er_xM_{1-x}Al_3(BO_3)_4$ with various concentrations of M=Yb,Gd. As described in Section 2, all of these crystals were grown by us. Yb and Gd were used as both sensitizers and diluent atoms since Er is known to suffer from both weak absorption bands and cross-relaxation. Yb^{3+} transfers its energy mostly to the lower levels of Er^{3+} (Ref. 5), thereby improving the three-level operation of the Er^{3+} eyesafe laser. The role of Gd^{3+} as a sensitizer for Er^{3+} is not understood yet, although Gd^{3+} is used to sensitize Tb^{3+} luminescence. Both Yb^{3+} and Gd^{3+} are used also as diluent atoms to decrease cross-relaxation between Er^{3+} ions. We found that especially in the case of $LiEr_xP_4O_{12}$, Gd^{3+}_{1-x} increased the luminescence. At present, the effects of both of these sensitizer ions are being studied to improve the properties of Er^{3+} eyesafe emission.

Figure 11 shows transmission spectra from each stoichiometric material. We used a representative sample from each group which did not contain any sensitizers (for clarity purposes) and which had large suitable areas for absorption measurements. These absorption spectra compare well with those in published reports (Refs. 6, 7, 8).

The emission spectra of these crystals were studied both in the visible and near-infrared regions of the spectrum. A PbS detector attached to a monochromator was used to detect emission in the eyesafe region. We have found that both the visible and infrared emissions from the $Er_xM_{1-x}Al_3(BO_3)_4$ samples were extremely weak, and that our PbS detector was not able to detect any eyesafe emission from this material. Also, we have found that the emission from pure $LiErP_4O_{12}$ samples was relatively weaker than that from similar samples containing Yb and Gd. This latter behavior was not observed in ErP_5O_{14} although previous studies showed that these two materials effect the luminescent ion in similar fashion. Figure 12 shows the emission spectra of an ErP_5O_{14} and a $LiErP_4O_{12}$ sample in the eyesafe region. As expected, both the location and shape of these lines for the two materials are about the same.

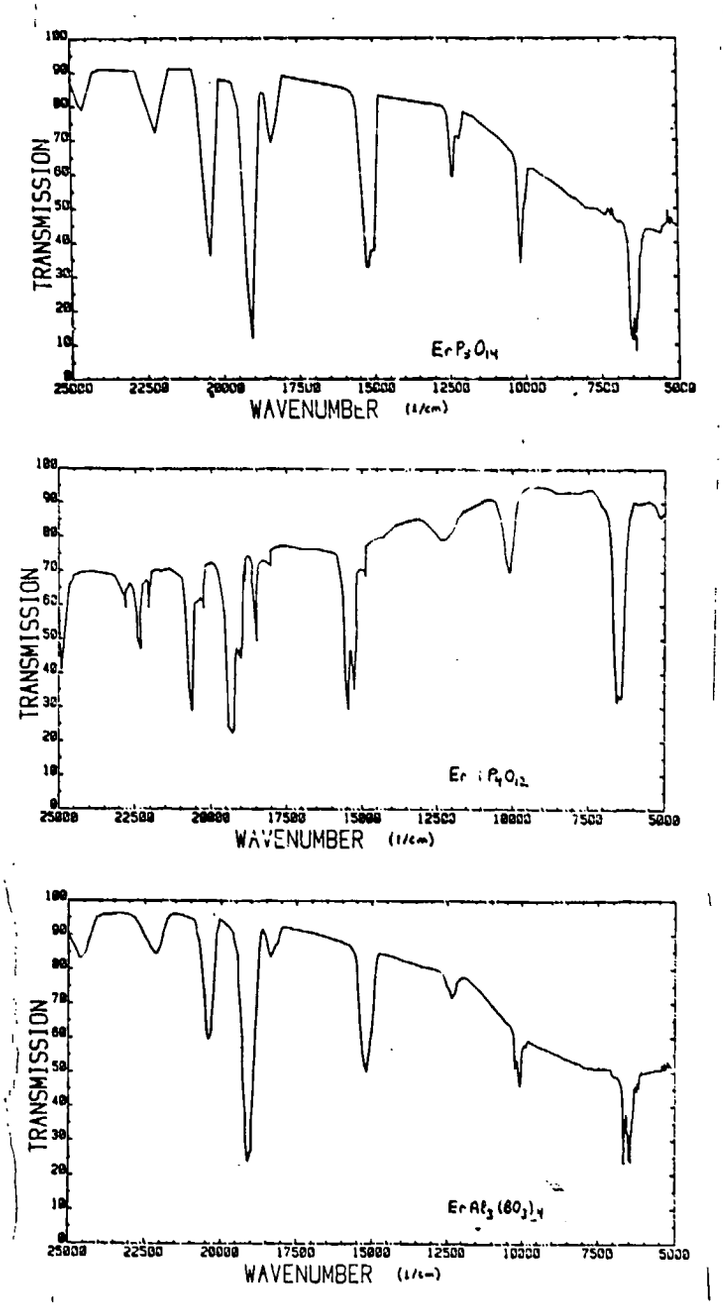
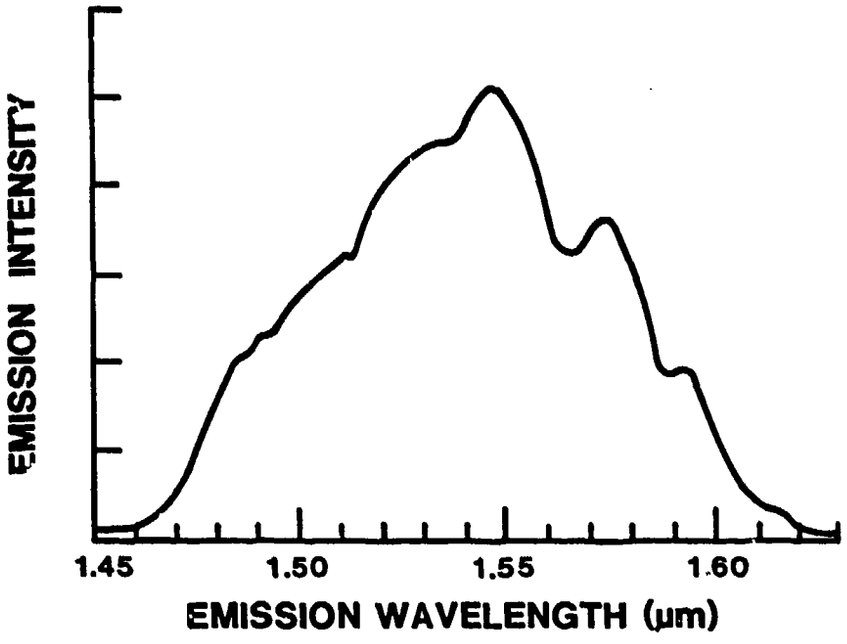
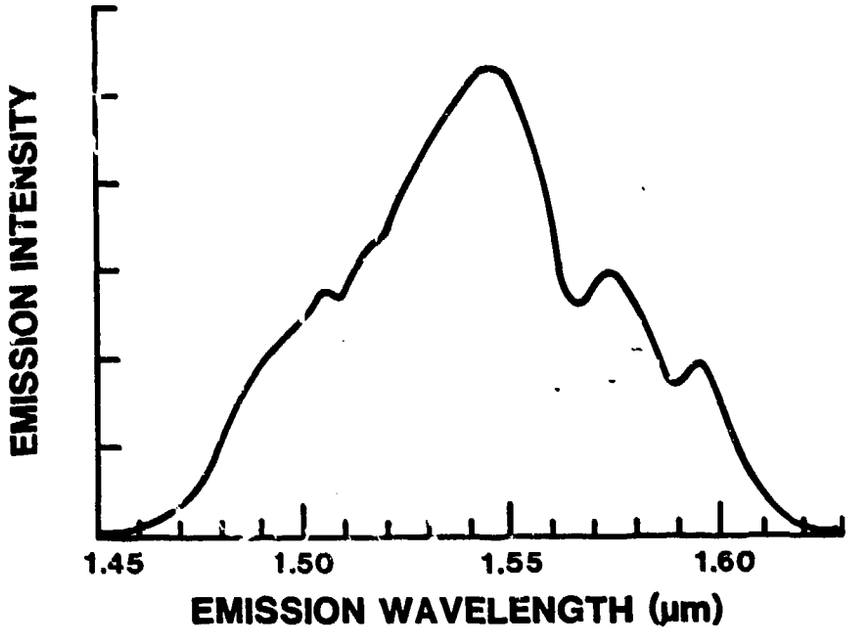


Figure 11: Transmission spectra of ErP_5O_{14}, $LiErP_4O_{12}$, and $ErAl_3(BO_3)_4$ in the visible and near infrared. Note absorption line near 1500 nm corresponding to three-level laser transitions of Er^{3+} between $^4I_{13/2}$ and $^4I_{15/2}$ multiplets.



(a) ErP_5O_{14}



(b) $LiErP_4O_{12}$

Figure 12: Emission spectra of ErP_5O_{14} and $LiErP_4O_{14}$ in eyesafe region.

It is known that Er^{3+} has two different transitions which result in emission around 1.50 - 1.60 μm. The upper one of these transitions is between the $^4S_{3/2}$ and $^4I_{9/2}$ which results in a four-level laser operation. This was the case reported in YLF where Er^{3+} emits at 1.73 μm (Ref. 9). The lower transition is from $^4I_{13/2}$ to the ground multiplet $^4I_{15/2}$, and because of three-level laser operation its threshold is higher. Our initial experiments on the lifetime of the eyesafe emission from our samples showed that most of the emission is originating from the lower transition which has a much longer lifetime than the upper transition.

In order to evaluate the materials for laser applications, we computed emission cross sections and obtained lifetimes of the upper four-level-type transition (Refs. 6, 7). The computations were only for phosphate crystals since data for borates were not available. The results are listed in Table 3. As shown, the lifetimes and emission cross-sections for the upper four-level, eyesafe laser transition are rather low. This is reflected in high cw threshold pump densities given in the last row. Although it may be a problem to obtain such cw power densities, pulsed operation seems possible.

Table 3: Spectroscopic Evaluation of Laser Properties of ErP_5O_{14} and $LiErP_4O_{12}$ Crystals.

Parameter	Crystal	
	ErP_5O_{14}	$LiErP_4O_{12}$
Transition Levels	$^4S_{3/2}$ ---> $^4I_{9/2}$	$^4S_{3/2}$ ---> $^4I_{9/2}$
Lifetime, τ (sec) *	1.2×10^{-6}	1.1×10^{-6}
Radiative Transition Rate, A, (sec^{-1}) *	74	45
Peak Emission Cross Section, σ_e, cm^2 **	2×10^{-21}	1.2×10^{-21}
Threshold Parameter $(\sigma_e \tau)^{-1}$ (cm^{-2} sec^{-1})	4.2×10^{26}	7.7×10^{26}
CW Threshold Power Density, A_T/A $W/(cm)^2$ ***	1.6×10^6	2.9×10^6

(*) Refs. (6) and (7)

(**) $\sigma_p \cong \frac{\lambda^4 A}{8\pi c n^2 \Delta\lambda}$, $\lambda = 1.55 \times 10^{-4}$cm, $\Delta\lambda \cong 7 \times 10^{-6}$cm, $n \cong 2$

(***) $\frac{A_T}{A} \cong \frac{h\nu_p}{\sigma_e \tau} \left(\frac{L + T}{2}\right)$, where $\lambda_p = 530$ nm, $L + T \cong 0.02$ (see Ref. 10)

5. PLANS FOR NEXT QUARTER

The following efforts will be continued during the next concluding quarter. A summary of these results will be included in the final report.

a. Continue growth of NPP crystals and fabrication of laser rods.

b. Continue growth of crystals of $Er_xGd_{1-x}P_5O_{14}$, $Er_xGd_{1-x}Li(PO_3)_4$ and $Er_xGd_{1-x}Al(BO_3)_4$.

c. Study spectroscopic properties of Er^{3+} stoichiometric materials for lasing applications.

d. Try to lase Er^{3+} crystals with flash lamp pumping.

e. Increase efficiency of Q-switched output from NPP laser.

f. Study effects of high pulse repetition on output energy of NPP laser.

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